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14. ABSTRACT

Thin film silicon has many useful purposes. Among the applications are solar cells and thin film transistors.This project involves a new and potentially lower cost method to produce thin silicon films. The method is called metal induced growth (MIG). A thin catalyst metal layer deposited on a foreign low cost substrate serves as the basis for growth of a nanocrystalline silicon thin film with thickness of 5-10 microns and preferred orientation of (220). The silicon deposition by magnetron sputtering on the heated substrate resulted in columnar structured grains having a diameter up to about 0.5 microns. Schottky barrier solar cells fabricated on these films gave a photocurrent of about 5 mA/cm2 and open circuit voltage of 0.25 volts. A modified process gave NiSi crystalline nanowires with length up to 10 microns and diameter of about 50 nm.

15. SUBJECT TERMS

Thin film silicon, solar cells, thin film transistors, nanowires, metal induced growth

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FINAL PERFORMANCE REPORT
TO
AIR FORCE OFFICE OF SCIENTIFIC RESEARCH

"Metal Induced Growth of Si Thin Films and NiSi Nanowires"

FA9550-07-1-0034

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Table of Contents

Summary

This project involves a study of metal induced growth (MIG) to produce lower cost thin films of silicon on foreign substrates. MIG is a modification of metal induced crystallization (MIC) in which an amorphous silicon film is crystallized in a follow-on high temperature step. MIG has the advantage of using a one step and lower temperature process which is thus a lower cost alternative to MIC.

In MIG, a suitable substrate is first coated with a 40 nm thick layer of catalyst metal. This is followed by DC magnetron sputtering of doped Si from a 2 inch target with the substrate heated to about 625 ^{o}C. Ni, as the catalyst metal, first forms a nickel silicide nanocrystalline (nc) layer which then serves as the template for the following nc-Si film which results as the Ni is consumed during the first stages of Si deposition. The Si film is typically 5-10 microns in thickness with crystallites up to a micron in size. The top layer of the Si film still contains too much Ni residue. This has been improved by using a Co/Ni layered catalyst in which the Co confines more of the Ni to the substrate interface. This gives a higher purity Si film. The films were evaluated using scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), X-ray diffraction (XRD), transmission electron microscopy (SEM) and Auger electron spectroscopy (AES). The films have a predominant (220) orientation. Schottky photovoltaic diodes were fabricated to examine the potential of the films for solar cell application. Photocurrent levels of about 5 mA/cm^2 and open circuit voltage of about 250 mV were obtained. The current level was as expected but open circuit voltage lower than the desired 550 mV due to an excessive leakage current. This must be resolved in future work.

In an effort to reduce processing temperature, the MIG process was modified by using Al and Co/Al as the catalyst metal. With Al, the process can be lowered to about 325 ^{o}C which would result in a much lower cost to form thin film Si. With the Al catalyst, the process is a eutectic formation of the Si thin film rather than a silicide process as with the Ni catalyst. All of the above studies were conducted with the result that the films were inferior to the ones produced when using the Ni catalyst.

During these studies, modification of the deposition procedures with the Ni catalyst results in nanowire growth. The nanowires were crystalline NiSi with a length up to 10 microns and a diameter of about 50 nm. Dense arrays of nanowires have been formed which can give improved photon absorption in photovoltaic applications. Nanowires have also been grown between two metallic metal pads to demonstrate the application as conductors of electrical current related to nanostructures. NiSi is an excellent electrical conductor and extremely stable as well.

I. Goals and Objectives

With the escalating energy crisis and subsequent desire for alternative energy sources, solar energy has increasingly gained considerable interest . Solar energy harnessed through photovoltaics is an area that has been studied and, to a minor extent, employed for several decades. The chief impediment to the widespread implementation of this technology has been a matter of economics and the high cost compared with other, more established sources of energy. Most of these efforts in terrestrial photovoltaics have been directed toward wafer-based single crystal Si solar cells. While this technology is able to achieve relatively high efficiencies and will likely enjoy success through the next couple of years, the inability to significantly reduce the cost and ease of manufacturing has driven researchers to explore other options. One of these options involves the utilization of thin films [1].

Thin films are a much less expensive alternative to ingot wafers. Thin films, by nature, require far less material, and therefore, are characteristically less expensive. The cost of manufacturing these thin films may be further reduced with the assistance of lower processing temperatures. As the processing temperatures are further decreased, it is eventually possible to employ inexpensive substrates, such as plastics and glass, which additionally reduce cost.

While there are many materials being researched for thin film solar cells, the use of thin film Si has the advantage of near-limitless abundance, as well as, familiarity with established microelectronic processes. Typically, thin film Si solar cells use either microcrystalline Si (µc-Si) or amorphous Si (a-Si) because they have much higher absorption coefficients than single crystal Si, allowing for thinner films. Although it may be more difficult to achieve, µc-Si is more desirable than a-Si due to its increased mobility. Thin film µc-Si is also a popular material for thin film transistors (TFT). These TFTs can be applied to numerous other applications, such active-matrix arrays for flat-panel displays. In all, while the efficiencies of thin film Si cannot yet compete with those of single crystal Si, the vast improvements in the cost and ease of manufacturing may already make it a viable alternative.

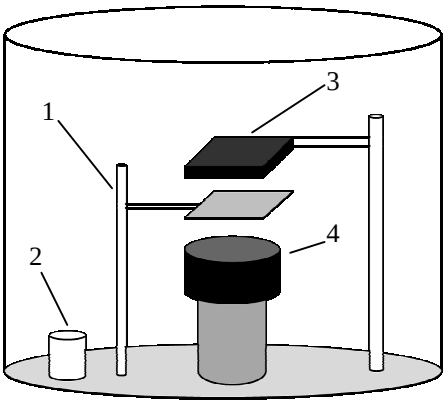

Figure 1. Schematic diagram of dc sputtering system: 1-Shutter, 2- Gas inlet, 3-Substrate holder, 4-dc magnetron and target

Metal-induced growth (MIG) is a process that is capable of producing thin films of µc-Si at relatively low temperatures [2]. It is similar to a more established process of crystallizing Si known as metal-induced crystallization (MIC) [3]. MIC is a two-step process where a-Si is first deposited then it is later converted to crystal form in an annealing step. MIG has the advantage of performing the deposition and crystallization in a single step. In conjunction with this, the

process simultaneously forms an ohmic back contact, which reduces the number of steps in subsequent device fabrication. When Si is sputtered from a target at an elevated temperature, a pre-deposited thin metal film behaves as a catalyst in crystallization of the incoming Si and forms a crystallized Si seed layer. As the sputtering process progresses, a continuous µc-Si film is produced.

II. Experimental

The MIG process began with a substrate of either oxide-coated Si or tungsten. The oxide coating was to prevent diffusion of the thin metal into the Si substrate and electrically isolate the MIG process, while tungsten was used to demonstrate the feasibility of the process on a foreign substrate. An initial thin metal layer was thermally evaporated on the substrate, which was then immediately transferred to the dc sputtering chamber. In this study, the process was first optimized using a combination of Ni and Co catalyst metals. Later, Al was substituted as a means to explore more effective metals and further lowering of processing costs. The thickness of metal layers was varied from 7.5 nm up to 70 nm. After the substrates reached the proper temperature, an n-type Si target was sputtered using a dc magnetron. The initial slow rate sputtering was then administered to form the seed layer. The rate was then increased in order to establish the µc-Si film. The resulting grains formed a columnar structure, which allowed for fewer grain boundaries through the vertical pathways of the film. For the Si substrates, a separate metallization was completed prior to sputtering to protect the back contact. The conductive tungsten, on the other hand, formed the back contact without the need for any additional processing. After MIG, the films were annealed in forming gas of 85% N_2 and 15% H_2 to relieve any stress in the film. Structural and material analysis was done on the completed films. X-ray diffraction (XRD) was used to confirm and compare the crystallization of Si with variations in deposition procedures. Scanning electron microscopy (SEM) was used to evaluate the surface and cross-sections of films, while energy dispersive x-ray spectroscopy (EDS) measured the atomic composition of films to determine the amount of metal throughout the film.

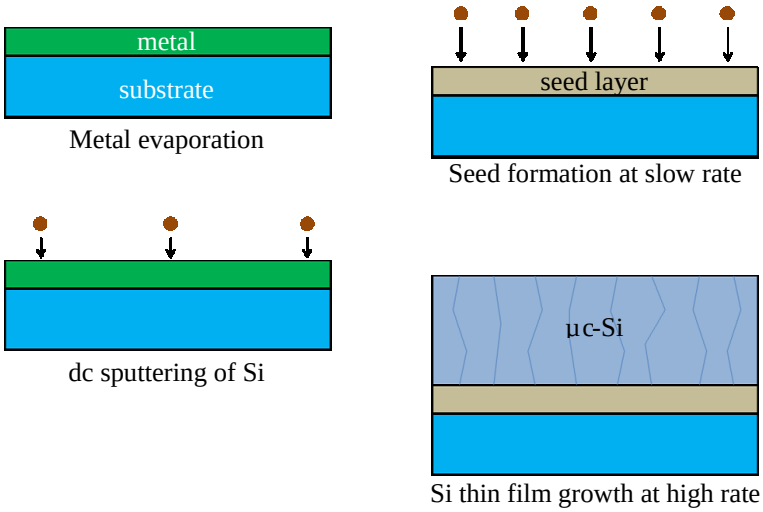

Figure 2. Process flow for MIG.

The physical mechanism by which crystallization occurs differs depending on the role and properties of the metal, and it is important to understand these differences. While nearly any

metal is capable of lowering the crystallization temperature of Si, certain metals become more efficient based on their interaction with Si. Metals in this process can generally be lumped into two different categories: compound-forming metals and eutectic metals. However, even within these categories, there can be distinct differences. With compound-forming metals, such as Ni and Cu, a silicide compound is initially formed. With Ni, this silicide precipitates through the Si and crystallizes it by the diffusion of Ni and Si atoms [4]. Typically, the crystallization process does not begin until the temperature reaches the point at which the most silicon-rich silicide is formed. In the case of Ni, this compound is $NiSi_2$. However, with Cu, the most silicon-rich compound is Cu_3Si. Therefore, because this is still a metal-rich silicide, this compound does not precipitate and instead behaves as a latent heat source, which provides a high enough heat density to activate the phase transition to crystallized Si [5]. On the other hand, eutectic metals do not form a silicide. Instead, Si atoms diffuse into the metal and form nucleation sites where Si grains will grow that eventually come into contact with each other and form a continuous film [6]. This process is dependent on the solubility of Si in the metal and typically starts at much lower temperatures than compound-forming metals [3].

Applications of MIG films were also explored and evaluated. Schottky photodiodes were fabricated to compare the changes in photovoltaic performance. The films were first cleaned with buffer HF, and ramped to 600 ^{o}C to form a native passivating oxide. A 10-nm film of Au was deposited to form the Schottky junction. The devices were measured under dark and one-sun illumination conditions. Solar cell parameters, such as open circuit voltage (V_{oc}), short circuit current density (J_{sc}), and fill factor (FF) were obtained and used to determine optimal deposition parameters and film quality.

III. Key Results

Extensive studies were conducted with metal catalysts to determine the effects of parameters in the MIG process on both the structural and electrical properties of the resulting films. The structural study can provide a good indication of the electrical properties and photovoltaic performance. While the metal is necessary for effective crystallization, an excess in the film can contaminate it and cause undesirable electrical results. Also, because the films are microcrystalline, grain boundary effects must be taken into account. Therefore, the ability to achieve larger grain sizes is desirable in order to offset these adverse effects [7].

3.1. Material and Structural Analysis of Films Using Ni and Co

The MIG process was first studied using metal layers of Ni, Co, or a combination of the two. At a temperature of 625 ^{o}C, these metals are very effective at crystallizing Si. However, the thickness of the metal film is an important factor. It was discovered that the lower limit of metal thickness is 7.5 nm. At this thickness, SEM studies revealed that, regardless of the metal used, it did not result in proper grain formation. However, as the metal thickness increased, the grain size increased. The largest grain sizes were 0.6 µm and were achieved using Ni alone, as seen in Figure 3. While the effects were similar with the other metals, Co alone achieved the smallest grains, and Co/Ni produced larger ones.

While this would indicate that Ni alone would be the best option, an atomic analysis study using EDS near the surface of the films painted a more complete picture. These results showed a strong presence of Ni in films using Ni alone. This was due to the diffusion of unconsumed Ni atoms, and the precipitation of the silicide. Films using Co and Co-coated Ni achieved much less metal contamination. In the case of Co-coated Ni, the Co was able to

provide a diffusion barrier for the Ni atoms. Therefore, films using Co-coated Ni were able to obtain high purity Si while utilizing the advantageous properties of large grain sizes provided by Ni.

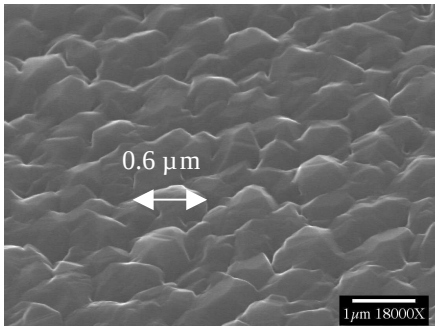

Figure 3. Large grain sizes achieved using Ni induced growth.

Another important parameter in the MIG process is sputtering rate. The sputtering rate is directly proportional to the sputtering power. While the sputtering power in the slow rate step was always 50 W, the subsequent high-rate, film-thickening step was varied from 150 W to 225 W. Lower power or rate allowed more time for Si atoms to adsorb to the surface of the film and therefore form large grains. Although a lower rate resulted in high quality films, the reduced processing speed might affect cost. In the EDS study, there was little difference in contamination levels with sputtering power. The Co did a sufficient job of providing a diffusion barrier regardless of sputtering rate.

3.2. Device Analysis of Films Using Ni and Co

Table 1. Effects of metal thickness to photovoltaic parameters.

Ni thickness [nm]	**Co thickness [nm]**	**J_{sc} [mA/cm^2]**	**V_{oc} [V]**
7.5	-	-	-
60	-	-	-
-	7.5	-	-
-	30	2.8	0.052
-	60	0.6	0.02
25	5	0.5	0.01
25	25	1	0.075
25	45	2.5	0.19
23	45	3.6	0.205
45	45	0.9	0.13

Once the Schottky devices were fabricated, measurements were performed under one-sun illumination, and the resulting changes in photovoltaic parameters were evaluated. The metal thickness was determined to play a crucial role. Table 1 details the results of various thicknesses. When Ni alone was used, devices displayed no rectification. This was likely due to the Ni contamination, which acted in shorting the device. Films using Co were able to achieve rectification, but the small grains hindered the V_{oc}. Carefully selecting the proper combination of Ni and Co thicknesses resulted in far better performance with nearly a fourfold increase in V_{oc}

over Co alone. Though the Co must be thick enough to prevent Ni diffusion, the ratio of Ni and Co thickness is also critical. The best results were obtained with Ni and Co thicknesses of 23 nm and 45 nm, respectively.

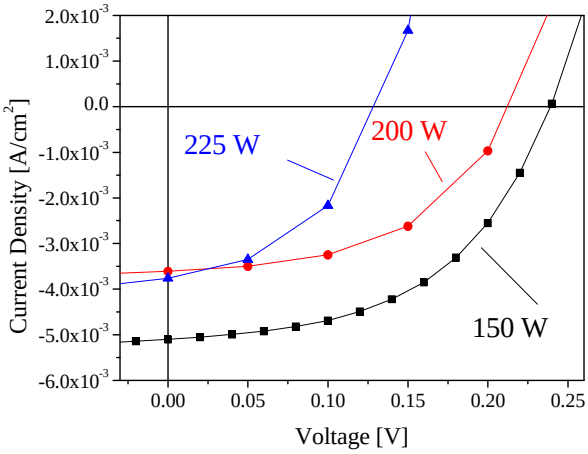

Figure 4. Effect of sputtering rate on photovoltaic performance.

Like the study of metal thickness, the photovoltaic results of changes in sputtering rate also agreed with the results of the materials analysis. Figure 4 shows the results obtained from these changes using the previously-obtained, optimal thicknesses. With decreasing power, and consequently increasing grain size, the photovoltaic performance improved. When the power was decreased from 225 W to 200 W, only the V_{oc} increased. However, a decrease to 150 W provided improvements to both J_{sc} and V_{oc} parameters.

3.3. Evaluating the Intermediate Step in MIG

In order to better understand the physical process during MIG, structural analysis with XRD was conducted after the initial slow rate step and then again after the full MIG process was completed. This study was done using only Ni as the catalyst metal. In Figure 5a, the crystal structure indicated a strong peak for NiSi with smaller peaks for $NiSi_2$, demonstrating that the silicide layer had begun to fully form. This layer became the seed layer upon which the µc-Si would grow. When the MIG process was fully completed, the crystal structure in Figure 5b indicated that the silicide layer has given way to strong crystalline peaks of Si with (220) orientation being dominant. This confirmed the compound-forming mechanism of Ni, as well as, the true crystalline nature of the resulting films.

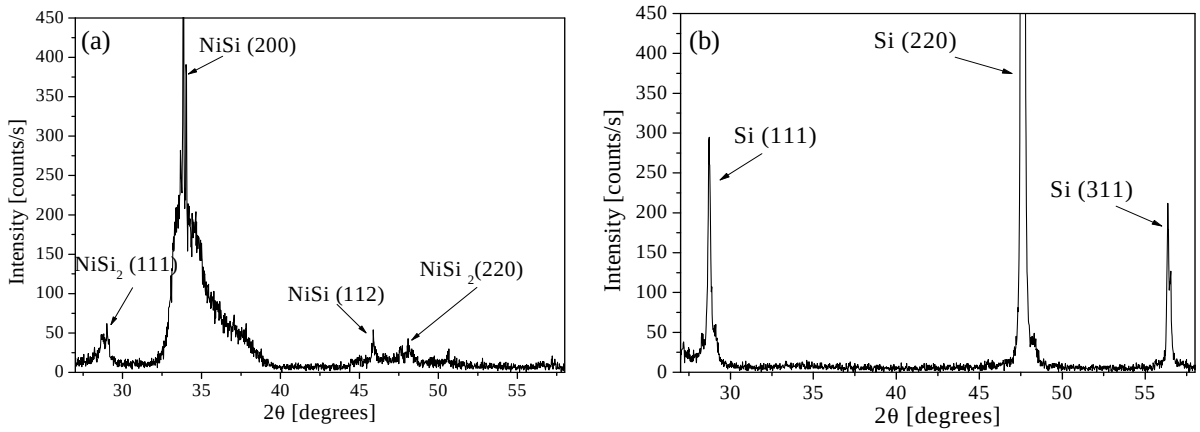

Figure 5. Crystal structure of films after (a) the slow rate step and (b) the complete MIG process.

3.4. Material and Device Analysis of Films Using Al

While the combination of Ni and Co has yielded good results, the process cannot be effectively carried out at temperatures below 575 ^{o}C due to the silicide nature of Ni. The crystallization process with Al has been reported to start at temperatures as low as 300 ^{o}C [3]. If the MIG process were able to approach these temperatures, it would allow utilization of less expensive, foreign substrates, such as plastics. The lower thermal budget in conjunction with less expensive substrates would further reduce the manufacturing cost of MIG.

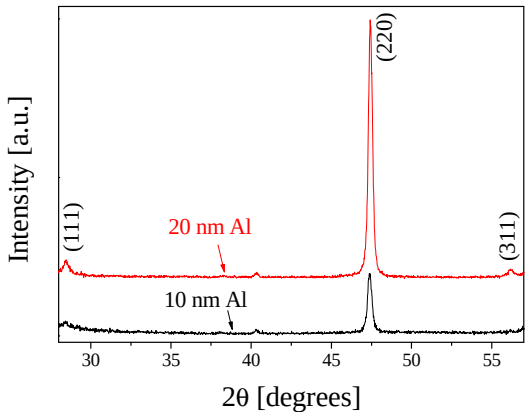

Figure 6. Large reduction in crystallization when 10 nm Al is used.

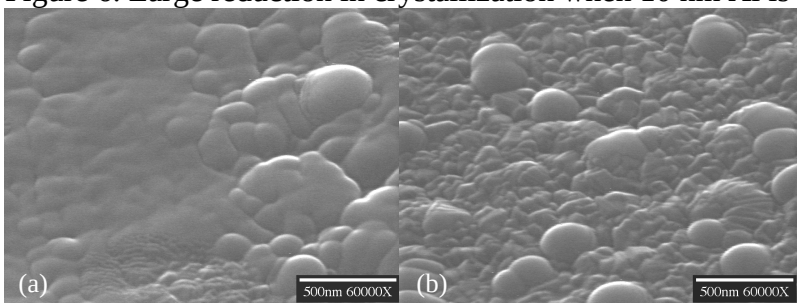

Figure 7. Similar observations to XRD at the surface of films with (a) 10 nm and (b) 20 nm Al.

Studies were conducted with films using Al as the catalyst metal. Similar to the studies with Ni and Co, structural analysis was first performed. In these studies the sputtering rates were

held constant at the values which obtained the best results in the previous studies. However, the thickness of Al and the deposition temperature were varied. In order to determine the critical Al thickness, the deposition temperature was first held at 525 °C. Analysis of XRD and SEM at the surface in Figures 6 and 7, respectively, demonstrated a weak crystallization at 10-nm Al. At 20 nm, the crystallization improved significantly. Therefore, the critical Al thickness was determined to be between 10-20 nm.

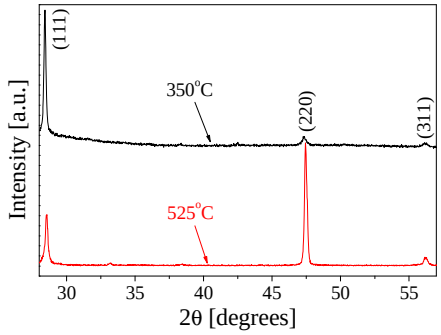

Figure 8. Crystal structure changes as the deposition temperature is decreased.

Next, the effect of deposition temperature was examined. Depositions were conducted at 525 °C and 350 °C with Al thickness of 70 nm. The XRD results in Figure 8 for the film at 525 °C showed similar crystalline Si peaks as before with a dominant peak at (220) Si. However, at 350 °C, the films observed a shift to (111) Si. While this indicated a change in the structure of the film, it confirmed that µc-Si was still obtained at a temperature of 350 °C.

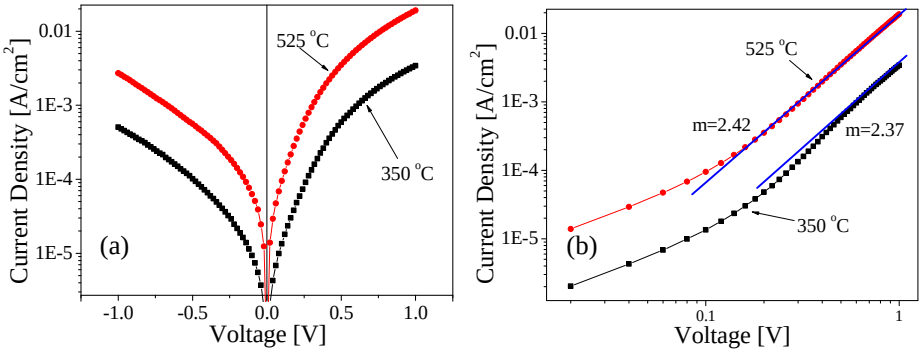

Figure 9. Electrical measurements illustrating SCLC with values of m greater than 2.

Schottky diodes were fabricated on these films to evaluate their electrical performance. The current-voltage measurements in Figure 9 showed that, regardless of temperature, the diodes exhibited strongly space-charge limited conduction (SCLC). SCLC is indicated by linearity in the log-log plot. The value of the slope for both devices was greater than 2, which indicates that the SCLC is a result of an exponential distribution of trap levels [8]. These traps are likely Al atoms which have diffused into the film and formed recombination centers at the grain boundaries. The photovoltaic performance was also poor.

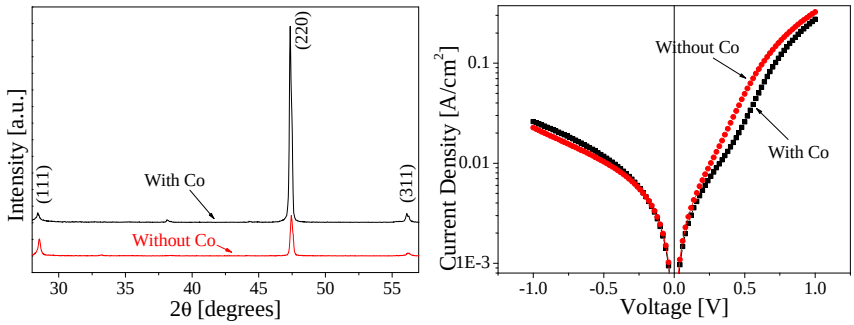

Figure 10. Changes in (a) XRD and (b) electrical measurements with the addition of Co.

Therefore, Co was employed in a similar manner to the Ni films. As in the Co/Ni case, the Co would behave as a diffusion barrier to the Al atoms. Structural results showed improved crystallinity in Figure 10a. Electrical measurements in Figure 10b also showed a shift to a junction-controlled behavior with the devices no longer behaving under SCLC. Because crystallization with Co does not occur under 600 ^{o}C, the increased crystallinity and improved diode performance can be attributed to increased confinement of Al. However, while there were significant improvements to the films, the photovoltaic performance was still poor. The best results obtained can be seen in Figure 11.

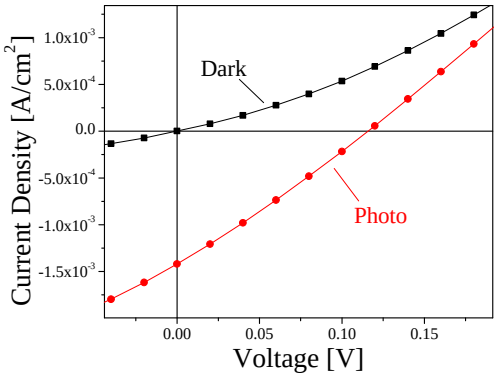

Figure 11. Best results obtained for MIG using Al.

Based on EDS studies, there is still a large amount of Al contamination even when Co is used. The mechanism for crystallization with Al is likely the debilitating factor. The Al is not consumed in a compound and is therefore free to diffuse through the film. Since Al is also a dopant in Si, this creates a heavily doped film which limits the generation of minority carriers.

3.5. Nanowire Formation through MIG Modification

Through modifications in the MIG process, it was also possible to grow nanowires. This was conducted using a Ni catalyst due to its high diffusion coefficient in Si. The modifications included temperature, Ni thickness, and sputtering rate. The temperature was 575 ^{o}C which is close to the crystallization temperature for Si films. This also required increased Ni thickness, 70 nm, and a decrease in sputtering rate, 20 W. This allowed for the migration of Ni into the Si, and the formation of a large array of crystalline NiSi nanowires. Individual nanowires were roughly 10 µm in length and about 50 nm in diameter.

These nanowires can be applied to numerous applications. A dense array of nanowires as seen in Figure 12a can serve as an absorber layer with increased optical absorption to improve solar cell efficiencies [9]. Nanowires were also grown between two electrical pads to form a nanobridge as in Figure 12b. They showed very good conduction and could serve as interconnects in nano-scale devices. By modifying the process further and substituting an Au catalyst, nanoblades in Figure 12c were formed which may have additional potential as textured absorber layers.

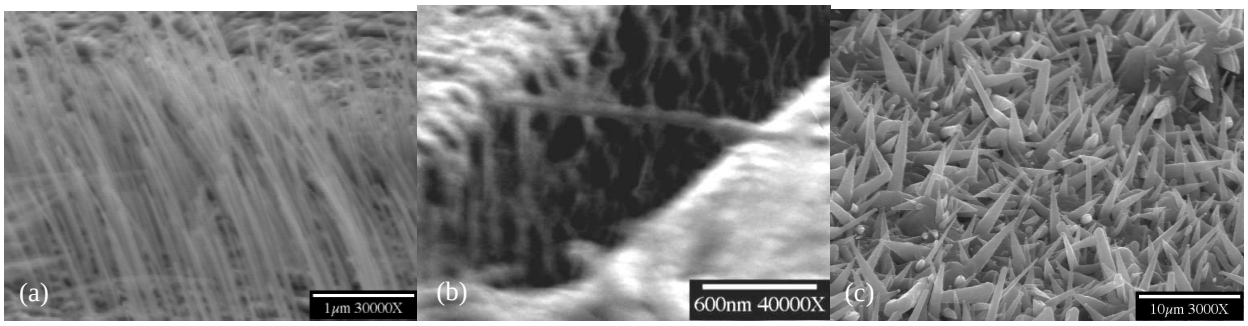

Figure 12. SEM images depicting (a) an array of nanowires, (b) nanobridge, and (c) nanoblades.

IV. Future Research Plans

1. *Post-processing of MIG Films*- Applications of MIG Si thin films are hindered by the small amount of metal impurity in the film due to the nature of the MIG process. For example, the Ni impurity can be removed using a mixture of H_2SO_4:H_2O_2, 10:1. Also, the nanocrystalline nature of the film introduces a large density of grain boundaries. These can be negated using H-passivation or a combination of H and Li passivation.

2. *Use of Other Catalyst Metals*- Thus far we have focused upon catalysts of Ni, Co/Ni, Al and Co/Al. The Ni process requires 550-625 ^{o}C. The Al process can be conducted at 300-550 ^{o}C but suffers from an inferior film. It is desirable to form thin-film Si at a lower temperature for economic reasons but electrical quality must be maintained. Other metal catalysts, such as Cu, will be explored

3. *Fabrication of Thin Film Transistors (TFTs)*-The past focus has been on photovoltaic devices for lower cost energy conversion. Another application is that of TFT's for flat panel displays. Studies of this application will be conducted with the added benefit of making measurements of field effect mobility of the films.

4. *Nanowire Growth*- Growth of nanowires will be resumed with the intent to apply arrays of nanowires to improve solar cell efficiency. Nanowires thus far have been a Ni:Si formulation. These are good conductors and may serve as antireflecting photon absorbers to greatly enhance current generation of solar cells.

5. *Zinc Oxide Over MIG Silicon*- We have been studying the formation of ZnO films by RF sputtering. Part of this study deals with the formation of ZnO/Si heterojunctions. These structures have recently been shown to give a significant photocurrent gain. Thus, ZnO/Si

can be explored for low cost arrays of sensitive photodetectors. Also, ZnO/MIG-Si can serve as a solar cell structure with improved photon absorption and higher solar conversion efficiency.

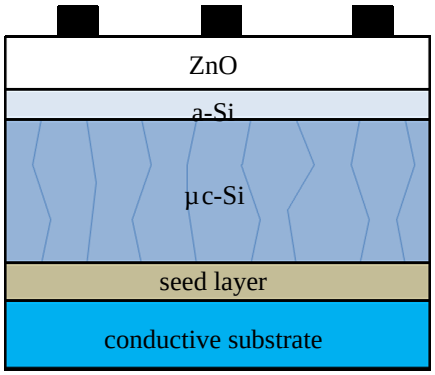

Figure 13. Proposed device structure for thin film ZnO/Si solar cells.

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VI. Conferences Based on this Work

1. Peter Mersich, S. Verma, A. Gaeta and Wayne Anderson, "Effects of Nickel Thickness on Metal-Induced Growth of Silicon Films for Heterojunction Solar Cells", 33rd IEEE Photovoltaics Specialists Conference, San Diego, May 12-16, 2008.

2. Peter Mersich, Shubhranshu Verma, Wayne Anderson and Rossman Giese Jr., "Effects of Deposition Parameters on the Structure and Photovoltaic Performance of Si Thin Films by Metal Induced Growth, Materials Research Society Meeting, Boston, Dec 1-5, 2008.

3. Peter Mersich, Ting-Fang Yen and Wayne Anderson, "Hetero-Interface Modification in Thin Film ZnO/Si Solar Cells, 34th IEEE Photovoltaic Specialists conference, Philadelphia, June 7-12, 2009.

4. Peter Mersich, Wayne Anderson and Rossman Giese Jr., "Lower Temperature Si Thin Films by Metal Induced Growth for Lower Cost Solar Cells", Materials Research Society Meeting, Boston, Nov 30- Dec 4, 2009.

VII. Publications Based on this Work

1. Joondong Kim*, Wayne A. Anderson, "Metal Silicide-mediated Microcrystalline Silicon Thin Film Growth for Photovoltaics", **Solar Energy Materials and Solar Cells** , **91,** 534, (2007).

2. Joondong Kim, C-S Han, Y C Park and W. A. Anderson, "Three-Dimensional Crystalline Si Film Growth by the Ni Silicide Mediation", **Appl. Phys. Lett., 92**, 501-503 (2008).

3. Joondong Kim, E-S Lee, C-S Han, Y Kang, D Kim and W. A. Anderson, "Observation of Ni Silicide Formations and Field Emission Properties of Ni Silicide Nanowires", **Microelectronic Engineering**, **85,** 1709-1712 (2008).

4. Joondong Kim and Wayne A. Anderson, "Metal Silicide-Templated Growth of Quality Si Films for Schottky Diodes", **Thin Solid Films**, in press, (2010).

VIII. Thesis Based on this Work

Shubhranshu Verma, " **Optimizing the Metal-Induced Growth of Microcrystalline Silicon for Solar Cells",** M.S. Thesis, University at Buffalo, (2008).

NOTE: Joondong Kim is a former Ph.D. student and is now a collaborator. Peter Mersich is scheduled to complete his **dissertation** in 2010.